EXPERIMENTAL WORK TO SHOW THE DIFFERENCES BETWEEN THREE-STAGE DEHYDRATION AND ONE-STEP DEHYDRATION.

1. LABORATORY PROCEDURES

THREE-STAGE procedure:

Rose flower No. 1

In a bottle of 300 ml of capacity, put a rose flower (weighing between 8 and 12 grams), and add a first mixture consisting of 176 ml of ethanol and 44 ml of water. Close the bottle and place in a water bath at 50 degrees Celsius for one hour.
Then let cool and drain the liquid into a glass labeled "First Mixture", stop draining the rose flower inside the bottle upside down for five minutes. Determine the water content of the first mixture outgoing, by the Karl-Fisher method..
Then add a second mixture, consisting of 198 ml of ethanol and 22 ml of water. Close the bottle and place in a water bath at 50 degrees Celsius for one hour. Then let cool and drain the liquid into a glass labeled "Second Mixture", stop draining the rose flower inside the bottle upside down for five minutes. Determine the water content of the second mixture outgoing, by the Karl-Fisher method. Then add a third mixture, consisting of 220 ml of ethanol without water(*). Close the bottle and place in a water bath at 50 degrees Celsius for one hour. Then let cool and drain the liquid into a glass labeled "Third Mixture", stop draining the rose flower inside the bottle upside down for five minutes. Determine the water content of the third mixture outgoing, by the Kall-Fisher method. Finally dry the bottle with the flower, without cap, at 150 degres Celsius for two hours. If the content of water in the glass labeled "First Mixture" is minor than 25.2%(**) use this mixtures for dehydrating the next flower, but if the content of water in the "First Mixture" is more or equal than 25.2%(**), discard the liquid in the glass labeled "First Mixture", pour the liquid from the glass labeled "Second Mixture" to the glass labeled "First Mixture", pour the liquid from the glass labeled "Third Mixture" to the glass labeled "Second Mixture" and refill the glass labeled "Third Mixture" with 220 ml of ethanol without water(*).

(*) "without water" = from 99.3 to 100 Gay-Lusac degrees (oGL)

(**) 25.2% = water content of ethanol of 80 oGL by Karl-Fisher method.

Rose flower No. 2

In other bottle of 300 ml of capacity, put a new rose flower (weighing between 8 and 12 grams), and add the first mixture from the glass labeled "First Mixture". Close the bottle and place in a water bath at 50 degrees Celsius for one hour.
Then let cool and drain the liquid into the glass labeled "First Mixture", stop draining the rose flower inside the bottle upside down for five minutes. Determine the water content of the first mixture outgoing, by the Kall-Fisher method..
Then add the second mixture from the glass labeled "Second Mixture". Close the bottle and place in a water bath at 50 degrees Celsius for one hour. Then let cool and drain the liquid into a glass labeled "Second Mixture", stop draining the rose flower inside the bottle upside down for five minutes. Determine the water content of the second mixture outgoing, by the Karl-Fisher method. Then add the third mixture from the glass labeled "Third Mixture". Close the bottle and place in a water bath at 50 degrees Celsius for one hour. Then let cool and drain the liquid into a glass labeled "Third Mixture", stop draining the rose flower inside the bottle upside down for five minutes. Determine the water content of the third mixture outgoing, by the Karl-Fisher method. If the content of water in the glass labeled "First Mixture" is minor than 25.2%(**) use this mixtures for dehydrating the next flower, but if the content of water in the "First Mixture" is more or equal than 25.2%(**), discard the liquid in the glass labeled "First Mixture", pour the liquid from the glass labeled "Second Mixture" to the glass labeled "First Mixture", pour the liquid from the glass labeled "Third Mixture" to the glass labeled "Second Mixture" and refill the glass labeled "Third Mixture" with 220 ml of ethanol without water(*).

(*) "without water" = from 99.3 to 100 Gay-Lusac degrees (oGL)
(**) 25.2% = water content of ethanol of 80 oGL by Karl-Fisher method.

Repeat this procedure several times and record the amount of alcohol without water used and the amount of flowers that are obtained.

ONE STEP procedure:

Rose flower No. 1

In a bottle of 300 ml of capacity, put a rose flower (weighing between 8 and 12 grams), and add a mixture consisting of 220 ml of ethanol without water(*)

Close the bottle and place in a water bath at 50 degrees Celsius for three hours.

Then let cool, drain the liquid and determine the water content of the mixture by the Kall-Fisher method.

Finally dry the bottle with the flower, without cap, at 150 degres Celsius for two hours.

(*) "without water" = from 99.3 to 100 Gay-Lusac degrees (oGL)

Rose flower No. 2

In a bottle of 300 ml of capacity, put a rose flower (weighing between 8 and 12 grams), and add a mixture consisting of 220 ml of ethanol without water(*)

Close the bottle and place in a water bath at 50 degrees Celsius for three hours.

Then let cool, drain the liquid and determine the water content of the mixture by the Kall-Fisher method.

Finally dry the bottle with the flower, without cap, at 150 degres Celsius for two hours.

(*) "without water" = from 99.3 to 100 Gay-Lusac degrees (oGL)

Repeat this procedure several times and record the amount of alcohol without water used and the amount of flowers that are obtained.

2. EXPERIMENTAL DATA

TABLE 1.

THREE-STAGE DEHYDRATION DATA	FLOWER No. 1	FLOWER No. 2	FLOWER No. 3	FLOWER No. 4	FLOWER No. 5	FLOWER No. 6	FLOWER No. 7	FLOWER No. 8	FLOWER No. 9	FLOWER No. 10	FLOWER No. 11
Bottle weight (grams)	217.9	221.1	228.2	219.7	217.6	220.1	219.1	217.9	221	228.2	226.2
+ Bottle cap weight (grams)	232.8	236.4	243.5	234.7	232.4	235.1	234	232.9	236.3	243.6	241.1
+ Rose flower weight (grams)	244	247.2	254.7	245.5	244	246.3	245.2	242.7	245.7	255.2	255.8
+ 220 ml of first mixture weight (grams)	430.6	422.3	423.9	406.7	399.3	408.9	400.8	392.7	389.3	392.4	385.9
FIRST STAGE											
Initial %v/v ethanol in this mixture	81.45%	88.77%	86.55%	83.56%	81.56%	94.40%	91.90%	88.39%	85.67%	82.93%	81.54%
1 hour 50oC, then cooled and drained terminal water %w/w Karl-Fisher	26.86%	17.57%	21.11%	23.50%	26.13%	11.12%	15.54%	18.77%	22.11%	23.69%	28.04%
Weight, bottle+cap+drained rose flower (grams)	249	251.3	261	249.6	249.8	251.3	249.3	247.7	250.6	260.6	261.6
+ 220 ml of second mixture weight (grams)	426.4	421.2	428.1	415.5	413.7	414.6	409.3	406.6	408.1	417.1	416.3
SECOND STAGE											
Initial %v/v ethanol in this mixture	90.78%	98.92%	98.07%	96.74%	96.09%	97.91%	96.41%	95.85%	94.86%	93.87%	91.92%
1hour 50oC, then cooled and drained terminal water %w/w Karl-Fisher	14.86%	2.68%	4.63%	5.50%	7.70%	5.02%	5.81%	7.22%	8.50%	11.05%	12.90%
Weight, bottle+cap+drained rose flower (grams)	248.7	251.1	259.9	249.1	248.8	250.3	248.8	247.1	250.1	259.3	260.9
+ 220 ml of third mixture weight (grams)	418.9	422	429.4	415.6	414	420.8	417.8	413.6	414.9	421.7	420.4
THIRD STAGE											
Initial %v/v ethanol in this mixture	99.90%	99.90%	99.68%	99.52%	98.60%	99.90%	99.76%	99.29%	98.92%	98.31%	97.85%
1 hour 50°C, then cooled and drained terminal water %w/w Karl-Fisher	1.47%	0.45%	0.66%	1.92%	2.93%	0.33%	0.93%	1.52%	2.40%	3.12%	3.98%
2 hours 150°C											
Weight, bottle + dryed rose flower (grams)	220.5	223.8	231.2	222.5	220.5	222.9	221.8	220.5	223.2	230.9	229.8
Water %w/w in the flower ending third stage	1.13%	0.34%	0.48%	1.42%	2.20%	0.25%	0.71%	1.12%	1.84%	2.39%	3.01%
Amount of alcohol without water used (ml)	220	440	440	440	440	660	660	660	660	660	660
Amount of flowers that are obtained.	1	2	3	4	5	6	7	8	9	10	11

TABLE 2.

ONE-STEP DEHYDRATION DATA	FLOWER No. 1	FLOWER No. 2	FLOWER No. 3	FLOWER No. 4	FLOWER No. 5	FLOWER No. 6	FLOWER No. 7	FLOWER No. 8	FLOWER No. 9	FLOWER No. 10	FLOWER No. 11
Bottle weight (grams)	217.9	221.1	228.2	219.7	217.6	220.1	219.1	217.9	221	228.2	226.2
+ Bottle cap weight (grams)	232.8	236.4	243.5	234.7	232.4	235.1	234	232.9	236.3	243.6	241.1
+ Rose flower weight (grams)	243.7	247.9	254.4	246.1	243.7	246.0	245.5	243.0	247.8	255.0	251.7
+ 220 ml of first mixture weight (grams)	418	421.4	429.5	421	420.5	419.3	423.9	418.4	423.5	430.6	426.1
Initial %v/v ethanol in this mixture	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%
3 hours 50oC, then cooled and drained terminal water %w/w Karl-Fisher	4.94%	5.32%	5.19%	5.28%	5.19%	5.09%	5.19%	4.67%	5.22%	5.21%	4.92%
2 hours 150°C Weight, bottle + dryed rose flower (grams)	220.6	223.9	230.7	222.7	220.6	222.8	222.0	220.5	223.7	231.0	229.0
Water %w/w in the flower ending stage	3.72%	4.02%	4.00%	3.89%	3.81%	3.83%	3.88%	3.47%	3.99%	3.93%	3.62%
Amount of alcohol without water used (ml)	220	440	660	880	1100	1320	1540	1760	1980	2200	2420
Amount of flowers that are obtained.	1	2	3	4	5	6	7	8	9	10	11

TABLE 3

THREE-STAGE DEHYDRATION Gay-Lussac degrees

Flower No.	1	2	3	4	5	6	7	8	9	10	11
first mixture initial	81.45%	88.77%	86.55%	83.56%	81.56%	94.40%	91.90%	88.39%	85.67%	82.93%	81.54%
first mixture FINAL	78.60%	86.56%	83.56%	81.56%	79.28%	91.75%	88.24%	85.54%	82.73%	81.37%	77.62%
second mixture initial	90.78%	98.92%	98.07%	96.74%	96.09%	97.91%	96.41%	95.85%	94.86%	93.87%	91.92%
second mixture FINAL	88.77%	98.10%	96.67%	96.04%	94.40%	96.41%	95.80%	94.77%	93.80%	91.84%	90.36%
third mixture initial	99.90%	99.90%	99.68%	99.52%	98.60%	99.90%	99.76%	99.29%	98.92%	98.31%	97.85%
third mixture FINAL	98.97%	99.70%	99.55%	98.64%	97.92%	99.78%	99.35%	98.94%	98.28%	97.76%	96.41%

TABLE 4

ONE-STEP DEHYDRATION Gay-Lussac degrees

Flower No.	1	2	3	4	5	6	7	8	9	10	11
first mixture initial	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%	99.90%
first mixture FINAL	95.06%	94.68%	94.81%	94.72%	94.81%	94.91%	94.81%	95.33%	94.78%	94.79%	95.08%

TABLE 5

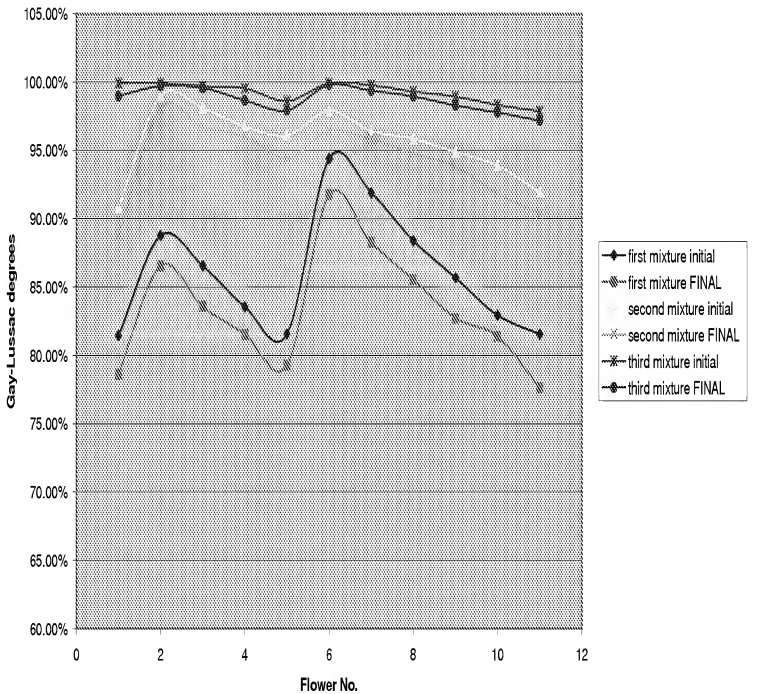
FLOWER WATER CONTENT (water %w/w)

	flower 1	flower 2	flower 3	flower 4	flower 5	flower 6	flower 7	flower 8	flower 9	flower 10	flower 11
THREE-STAGE INITIAL	76.79%	75.00%	73.21%	74.07%	75.00%	75.00%	75.89%	73.47%	76.60%	76.72%	75.51%
first stage FINAL	26.83%	17.56%	21.10%	23.47%	26.08%	11.12%	15.53%	18.75%	22.07%	23.64%	27.97%
second stage FINAL	14.85%	2.68%	4.63%	5.49%	7.69%	5.02%	5.81%	7.21%	8.48%	11.03%	12.87%
third stage FINAL	1.13%	0.34%	0.48%	1.42%	2.20%	0.25%	0.71%	1.12%	1.84%	2.39%	3.76%
ONE-STEP INITIAL	75.23%	75.65%	77.06%	73.68%	73.45%	75.23%	74.78%	74.26%	76.52%	75.44%	73.58%
one-step FINAL	3.72%	4.02%	4.00%	3.89%	3.81%	3.83%	3.88%	3.47%	3.99%	3.93%	3.62%

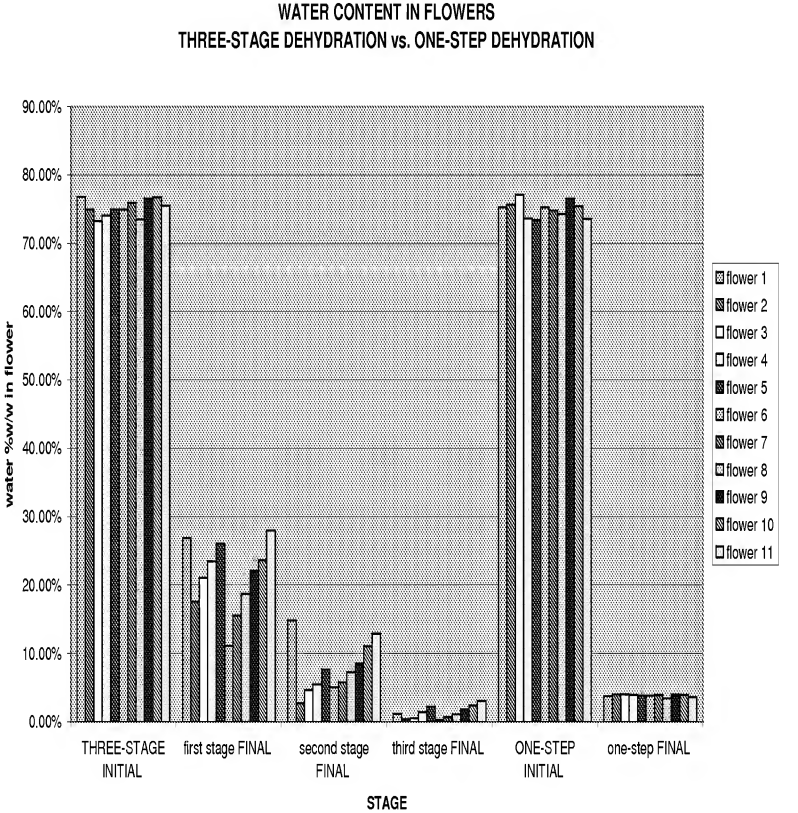
TABLE 6

SOLVENT CONSUMPTION

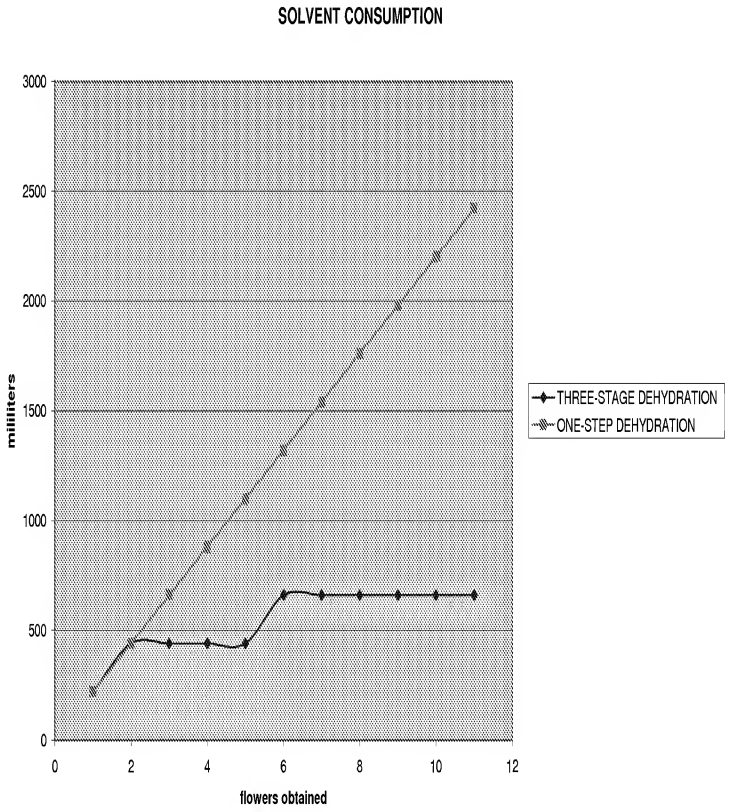
	1	2	3	4	5	6	7	8	9	10	11
THREE-STAGE DEHYDRATION	220	440	440	440	440	660	660	660	660	660	660
ONE-STEP DEHYDRATION	220	440	660	880	1100	1320	1540	1760	1980	2200	2420


Gay-Lussac degrees of initial and final mixtures
for first, second and third dehydration stages
105.00%
100.00%
95.00%
90.00%
85.00%
80.00%
75.00%
70.00%
65.00%
60.00%
Gay-Lussac degrees
0
2
4
6
8
10
12
Flower No.
first mixture initial
first mixture FINAL
second mixture initial
second mixture FINAL
third mixture initial
third mixture FINAL

Graphic 1.


WATER CONTENT IN FLOWERS
THREE-STAGE DEHYDRATION vs. ONE-STEP DEHYDRATION
water %w/w in flower
90.00%
80.00%
70.00%
60.00%
50.00%
40.00%
30.00%
20.00%
10.00%
0.00%
THREE-STAGE INITIAL
first stage FINAL
second stage FINAL
third stage FINAL
ONE-STEP INITIAL
one-step FINAL
STAGE
flower 1
flower 2
flower 3
flower 4
flower 5
flower 6
flower 7
flower 8
flower 9
flower 10
flower 11

Graphic 2.


SOLVENT CONSUMPTION
3000
2500
2000
1500
1000
500
0
milliliters
0
2
4
6
8
10
12
flowers obtained
THREE-STAGE DEHYDRATION
ONE-STEP DEHYDRATION

Graphic 3.

3. CONCLUTIONS

Three-stage dehydration shows unexpectedly better results than one-step dehydration, flowers are obtained having lower moisture content and using less alcohol.